Chapter NR 219

ANALYTICAL TEST METHODS AND PROCEDURES

NR	219.02	Purpose Applicability Definitions		NR	219.05	Identification of test procedures Alternate test procedures Laboratory certification or registration
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Note: A number of the references cited in this chapter are no longer in print. Copies of references which are out-of-print are available at any public library by inter-library loan.

NR 219.01 Purpose. The purpose of this chapter is to establish analytical test methods, preservation procedures, requirements for laboratories, and procedures applicable to effluent limitations for discharges from point sources as authorized by ss. 299.11 and 283.55 (1), Stats.

History: Cr. Register, August, 1976, No. 248, eff. 9-1-76; am. Register, April, 1986, No. 364, eff. 8-28-86; am. Register, June, 1986, No. 366, eff. 7-1-86; am. Register, April, 1988, No. 388, eff. 5-1-88; corrections made under s. 13.93, (2m) (b) 7., Stats., Register, November, 1996, No. 491.

NR 219.02 Applicability. (1) The procedures prescribed herein shall, except as provided in s. NR 219.06, be used in the determination of concentrations and quantities of pollutant parameters as required for:

- (a) An application submitted to the department for a permit under ch. 283, Stats.
- (b) Reports required to be submitted by dischargers in accordance with the conditions of issued permits.
- (2) Section NR 219.07 requires that laboratories conducting tests under this chapter be certified, registered, or approved under ch. NR 149, HSS 157 or 165.

History: Cr. Register, August, 1976, No. 248, cff. 9-1-76; am. Register, April, 1986, No. 364, cff. 8-28-86; am. (1) (intro.), Register, June, 1986, No. 366, cff. 7-1-86; correction in (1) (a) made under s. 13.93 (2m) (b) 7., Stats., Register, November, 1996, No. 491.

NR 219.03 Definitions. As used in this chapter:

- (1) "EPA" means the U.S. environmental protection agency.
- (2) "Department" means the department of natural resources. History: Cr. Register, August, 1976, No. 248, eff. 9-1-76; am. (I), (2), (3) and (4m), Register, January, 1978, No. 265, eff. 2-1-78; r. and recr. Register, June, 1986, No. 366, eff. 7-1-86; r. and recr. (1), r. (3) and (4), Register, November, 1992, No. 443, eff. 12-1-92.

NR 219.04 Identification of test procedures. (1) ANALYTICAL TEST PROCEDURES. Parameters or pollutants, for which wastewater analytical methods are approved, are listed together with test procedure descriptions and references in tables A to E. Parameters or pollutants, for which sludge analytical methods are approved, are listed together with test procedure descriptions and

references in table EM. Metals samples digestion procedures and references are listed in table BM. The discharge values for the listed parameters shall be determined by one of the standard analytical test procedures identified in a table under this subsection or by an alternate test procedure established under ss. NR 219.05 and 149.12.

- (2) SAMPLE PRESERVATION PROCEDURES. Sample preservation techniques, container materials, and maximum allowable holding times for parameters identified in tables A to E are prescribed in table F. Sludge samples shall be preserved at the time of collection by cooling to 4° C where required. All samples requiring preservation at 4° C shall be cooled immediately after collection, and the required temperature maintained during shipping. Any person may apply for a variance from the prescribed preservation procedures applicable to samples taken from a specific discharge. Applications for variances may be made by letters to the regional administrator and shall provide sufficient data to assure that the variance does not adversely affect the integrity of the sample. The regional administrator will make a decision on whether to approve or deny a variance within 90 days of receipt of the application.
- (3) TEMPERATURE REPORTING PROCEDURES. Samples cooled with ice packs or not in direct contact with ice during shipping shall be cooled to 4° C prior to shipping, and a temperature blank shall be submitted with the samples. Samples cooled during shipping with ice packs may not be recorded as received on ice. Samples may be recorded as received on ice only if solid ice is present in the cooler at the time the samples are received. If the samples are not received on ice, the laboratory shall record one of the following at the time of receipt:
 - (a) The temperature of an actual sample.
- (b) The temperature of a temperature blank shipped with the samples.
- (c) The temperature of the melt water in the shipping container.

 Note: Copies of the publications referenced in Tables A F are available for inspection at the offices of the department of natural resources, the secretary of state and the revisor of statutes. Many of these materials are also available through inter-library loan.

History: Cr. Register, June, 1986, No. 366, eff. 7–1–86; r. and recr. Tables B and E, Register, April, 1988, No. 388, eff. 5–1–88; am.; r. and recr. Tables A to F, Register, November, 1992, No. 443, eff. 12–1–92; am. (1), am. Tables A to F, Register, April, 1994, No. 460, eff. 5–1–94; am. (1) and (2), Tables A to F, cr. (3), Register, February, 1996, No. 482, eff. 3–1–96.

Table A
List of Approved Biological
Test Procedures For Wastewa-

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Table A List of Approved Biological Test Procedures For Wastewater

Parameter and Units	Method ¹	EPA	Standard Methods 18th Ed.	USGS	WDNR
Bacteria:			* * * * * * *		
Coliform (fecal) number per 100 mi	MPN, 5 tube, 3 dilution; or, membrane filter (MF) ² , single step.	p132 ³ p124 ³	9221E 9222D	B-0050-85 ⁴	
2. Coliform (fecal) in presence of chlorine number per 100 ml	MPN, 5 tube, 3 dilution; or MF, single step ⁵	p132 ³ p124 ³	9221E 9222D		

Table A
List of Approved Biological
Test Procedures For Wastewa-

Table A List of Approved Biological Test Procedures For Wastewater

ter

Parameter and Units	Method ¹	EPA	Standard Methods 18th Ed.	USGS	WDNR
Bacteria:	.3*			, , , , , , , , , , , , , , , , , , , ,	:
3. Coliform (total) number per 100 ml	MPN, 5 tube, 3 dilution; or, MF ² single step or two step	$\frac{p114^3}{p108^3}$	9221B 9222B	B-0025-85 ⁴	
4. Coliform (total) in presence of chlorine, number per 100 ml	MPN, 5 tube, dilution; or, MF ² with enrichment.	p114 ³ p111 ³	9221B 9222B+B.5c	• .	en e
5. Fecal strepto-cocci, number per 100 ml	MPN, 5 tube, 3 dilution; MP ² , or Plate count	p136 ³ p136 ³ p143 ³	9230B 9230C	B-0055-85 ⁴	1811 1414 1411 1114 1511 1114
Enteroviruses: 6. Enteroviruses in water, plaque forming units per liter.	Absorption, elution, and organic flocculation, followed by: Plaque assay (cell culture infectivity) Identification	Ch. 6 ⁶ Ch. 9 ⁶ Ch. 10 ⁶ Ch. 12 ⁶	9510B,C,D,E 9510G 9510G 9510G		
7. Enteroviruses in sludge, plaque forming units per liter.	Beef extract elution, and organic flocculation, followed by: Plaque assay (cell culture infectivity) Identification	Ch. 7 ⁶ Ch. 9 ⁶ Ch. 10 ⁶ Ch. 12 ⁶	9510F 9510G 9510G 9510G		Parameter (Control of Control of
Mutagenicity: 8. Mutagenicity (revertants per	Ames test, test strains TA97,	Note 7			
liter)	TA98, TA100, and TA102.	11010 7			
Acute and Chronic Toxicity: 9. Toxicity, acute, fresh water organisms, percent effluent 10	Ceriodaphnia, 48-h static- renewal mortality. Fathcad minnow, 96-h static- renewal mortality, or 96-h flow-through mortality.				8
10. Toxicity, chronic, fresh water organisms, percent effluent. 10	Fathead minnow larval survival and growth. Ceriodaphnia survival and reproduction.				.8

¹ The method used must be specified when results are reported.

² A 0.45 µm membrane filter (MF) or other port size certified by the manufacturer to fully retain organisms to be cultivated and to be free of extractables which could interfere with their growth.

³ Bordner, R.H., and J.A. Winter, eds. "Microbiological Methods for Monitoring the Environment, Water and Wastes", United States Environmental Protection Agency, EPA-600/8-78-017, 1978. Available from ORD Publications, CERI, U.S. Environmental Protection Agency, 26 W. Martin Luther King Drive, Cincinnati, Ohio 45268.

⁴ Britton, L.J., and P.E. Greeson, eds. "1988 Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples", edited by et al., U.S. Geological Survey, Techniques of Water—Resources Investigation (USGS TWRI), Book 5 chapter A4, Laboratory analysis, 1977. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

⁵ Because the MF technique usually yields low and variable recovery from chlorinated wastewaters, the Most Probable Number method will be required to resolve any controversies.

⁶ Berg, G., R.S. Safferman, D.R. Dahling, D. Berman, and C.J. Hurst, 1984. USEPA Manual of Methods for Virology. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA/600/4-84/013. (Chapter 9 revised January 1987; Chapter 10 revised December 1987; Chapter 12 revised May 1988; Chapter 7 revised September 1989).

Table B
List of Approved Inorganic Test Procedures for Wastewater

		.**				4 *
Parameter, Units & Methods	EPA1	SW-846 ^{11,7}	Standard Methods ^{2,2m}	ASTM ³	USGS ⁴	Other
1. Acidity, as CaCO ₃ , mg/L, Electrometric end point or phenolphthalein end point	305.1		2310 B(4a)	D1067-92		
2. Alkalinity, as CaCO ₃ , mg/L;						
Electrometric or colorimetric: Titration to pH 4.5, manual	310.1		2320 B	D1067-92	I103085	973.43 ⁵
Or automated	310.1		2320 B	D1007-92	1-1030-63	973,43
3. Aluminum, mg/L:						
Digestion ⁶ followed by:						
AA direct aspiration ^{6m} ,	202.1	7020	3111 D		I305I85	
AA furnace,	202,2 or		3113 B			
To decessors annual of interior	200.9 ^{1g}	60104	2120 D			
Inductively coupled plasma (ICP) ^{6m} ,	200.7 ^{1g}	6010A	3120 B			
Inductively coupled plasma-	200.8 ^{1g}	6020				
mass spectrometry (ICP-MS), Direct current plasma				D4190-82(88)		Note 36
(DCP) ^{6m} , or			0500 417			
Colorimetric (Eriochrome			3500-Al D			
cyanine R)		i				
4. Ammonia (as N), mg/L: Man- ual distillation ⁸ (at pH 9.5); Followed by	350.2		4500-NH ₃ B			973.49 ⁵ ,.
Nesslerization,	350.2		4500-NH ₃ C	D1426-89(A)	I-3520-85	973.46 ⁵
Titration,	350.2		4500-NH ₃ E	21.20 05(11)		2,12712
Electrode,	350.3		4500-NH3F&G	D1426-89(B)		
Automated phenate, or	350.1 ^{1m}		4500-NH ₃ H	• ,	I-4523.85	
Automated electrode						Note 9
5. Antimony, ug/L: Digestion ⁶ followed by:						
AA direct aspiration ^{6m} ,	204.1	7040	3111 B	2		
AA furnace,	200.91g	70 41	3113 B			
AA (gaseous borohydride),		7062				
Inductively coupled plasma ^{6m} , or	200.7 ^{1g}	6010A	3120 B			
Inductively coupled plasma-	200.8 ^{1g}	6020				
mass spectrometry						
6. Arsenic, ug/L:		-	,			
Digestion ⁶ followed by	206.5					
AA (gaseous hydride),		7061A	3114 B ³⁷	D2972-88(B)	I-3062.85	
AA (gaseous borohydride),	0050	7062	0110 P	Dagge Adver		
AA furnace,	206.2 or 200.9 lg	7060A	3113 B	D2972-88(C)	•	
Inductively coupled plasma6m,	200.7^{1g}	6010A	3120 B			
Inductively coupled plasma-	200.8^{1g}	6020				
mass spectrometry,			2500 + 0	D0070 00/A	T 20/0 95	
Or, colorimetric (SDDC)			3500-As C	D2972-88(A)	I-3060-85	

Williams, L.R., and J.E. Preston, eds. 1983. Interim Procedures for Conducting the Salmonella/Microsomal Mutagenicity Assay (Ames Test). Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Las Vegas, Nevada. EPA/600/4-82/068.

⁸ Compliance monitoring must be performed in accordance with the specifications in the "State of Wisconsin Aquatic Life Toxicity Testing Methods Manual, 1st Edition," Wisconsin Department of Natural Resources, 1996. This publication is available for inspection at the offices of the Department of Natural Resources, the Secretary of State, and the Revisor of Statutes. Copies are available from the Department of Natural Resource, Bureau of Integrated Science Services, P.O. Box 7921, Madison,WI 53707.

List o	of Approv	ed Inorganic		es for Wastewater		7.33
Parameter, Units & Methods	EPA ¹	SW-846 ^{11,7}	Standard Methods ^{2,2m}	ASTM ³	USGS ⁴	Other
7. Barium, mg/L: Digestion ⁶ followed by: AA direct aspiration ^{6m} , AA furnace, Inductively coupled plasma ^{6m} , Inductively coupled plasma— mass spectrometry, or Direct current plasma ^{6m}	208.1 208.2 200.7 ^{1g} 200.8 ^{1g}	7080A 7081 6010A 6020	3111 D 3113 B 3120 B	D4382-91	I-3084-85	Note 36
8. Beryllium, mg/L: Digestion ⁶ followed by: AA direct aspiration, AA furnace, Inductively coupled plasma, Inductively coupled plasma— mass spectrometry Direct current plasma, or Colorimetric (aluminon)	210.1 210.2 or 200.9 lg 200.7 lg 200.8 lg	7090 7091 6010A 6020	3111 D 3113 B 3120 B 3500–Be D	D3654-(88)(A) D3645(88)(B) D4190-82(88)	I-3095-85	Note 36
9. Biochemical oxygen demand (B mg/L: Dissolved Oxygen Depletion	OD ₅),		5210 B	F- 1:	I-1578-78 ¹⁰	
10. Boron, mg/L: Colorimetric (curcumin), Inductively coupled plasma, or Direct current plasma	212.3 200.7 ^{1g}	6010A	4500-В В 3120 В	D4190-82(88)	1-3112-85	Note 36
11. Bromide, mg/L: Titrimetric Ion Chromatography	320.1 300.01m	9056		D1246-82(88)(C)	I-1125-85	p.S44 ¹²
12. Cadmium-Total ⁶ , mg/L: Digestion ⁶ followed by: AA direct aspiration ^{6m} ,	213,1	7130	3111 B or C	D3557-90 (A or B)	I-3135-85 or I-3136-85	974.27 ⁵
AA furnace, Inductively coupled plasma ^{6m}	213.2 or 200.9 ^{1g} 200.7 ^{1g}	7131A 6010A	3113 B 3120 B	D3557-90(D)	I1472-85	The second of th
Inductively coupled plasma— mass spectrometry Direct current plasma ^{6m} , Voltametry ¹³ , or Colorimetric (Dithizone)	200.8 ^{1g}	6020	3500-Cd D	D4190-82(88) D3557-90(C)		Note 36
13. Calcium, mg/L: Digestion ⁶ followed by: Atomic absorption, Inductively coupled plasma, Direct current plasma, or EDTA titration	215.1 200.7 ^{1g} 215.2	7140 6010A	3111 B 3120 B 3500–Ca D	D511-92(B) D511-92(A)	I-3152-85	Note 36
14. Carbonaceous Biochemical oxygen demand (CBOD ₅), mg/L: with nitrification inhibitor ¹⁴		,	5210 B			

Parameter, Units & Methods	EPA ¹	SW-846 ^{11,7}	Standard Methods ^{2,2m}	ASTM ³	USGS ⁴	Other
15. Chemical oxygen demand (COD), mg/L: Closed reflux Titrimetric			5220 C or D			Notes 15&16 973.46 ⁵
Turneure	410.1 410.2		5220 B	D1252-88(A)	I3560 or I3562-85	973.40
Automated and manual Spectrophotometric	410.3 410.4 ^{1m}			D1252-88(B)	I-3561-85	
16. Chloride, mg/L:		.0252	. 4500 CL D	DS12 90/D)	I-1183-85	
Titrimetric (silver nitrate) or (Mercuric nitrate),	325.3	9253 9252A	4500-CI- B 4500-CI- C	D512-89(B) D512-89(A)	I-1184-85	973.51 ⁵
Colorimetric (ferricyanide), manual or automated, or	325.1 or 325.2	9250	4500-ClE	e _g e	I-1187-85 I-2187-85	
Ion chromatography	300.0 ^{1m}	9056				
17. Chlorine – Total residual,						
mg/L:	330.1		4500-Cl D	D1253-86(92)		•
amperometric, Starch End point direct	330.3 330.2		4500–Cl B 4500–Cl C			
Back Titration either end	JJU,2		4500-Cr C			
point ¹⁷ , or	330.4		4500-Cl F	**:		
DPD-FAS,	330.5		4500-CI G			37 40
Spectrophotometric, DPD; or Electrode			4500–Cl I	·		Note 18
18. Chromium VI dissolved, ug/L:						
0.45 micron filtration with:						
Extraction and atomic absorption,	218.4	7197	3111 A		I-1232-85	
Coprecipitation and atomic	210.4	1171	3111 A		1-1232-03	
absorption,		7195				
Differential pulse polarography,		7100				
Colorimetric (Diphenylcarbazide), or		7198 7196A	3500-Cr D	D1687-92(A)	I-1230-85	307B ¹⁹
Ion Chromatography	14 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	715011	3300 01 3	D1001		00,22
	218.6 ^{1g}			·		• .
19. Chromium, mg/L:		,		1 °		The second second
Digestion ⁶ (optional extraction) followed by:			1. 1.		•	25250 42
AA direct aspiration ^{6m} ,	218.1	7190	3111 B	D1687-92(B)	I3236-85	974.24 ⁵
AA chelation extraction,	218.3		3111 C	• •	5.3	aria de la companya
AA furnace,	218.2 or 200.9 ^{1g}	7191	3113B	D1687-92(C)		
Inductively coupled plasma6m,	200.7 ^{1g}	6010A	3120B			
Inductively coupled plasma— mass spectrometry,	200,8 ^{1g}	6020		.*		37 · · · · · · · · · · · · · · · · · · ·
Direct current plasma6m, or				D4190-82(88)	The second	Note 36
Colorimetric (diphenylcarbazide),			3500-Cr D		11.1	
20. Cobalt, mg/L:		*		* <u>, , , , , , , , , , , , , , , , , , ,</u>		
Digestion ⁶ followed by:	010 1	7100	. 2111 D /4 D)		T 2020 04	1.00
AA direct aspiration, AA furnace, or	219.1 219.2 or	7200 7201	3111 B (A or B) 3113 B	D3558-90(AorB) D3558-90(C)	1-3239-84	
Inductively coupled plasma, or	200.9 ^{1g} 200.7 ^{1g}	6010A	3120 B			
Inductively coupled plasma—	200.8 ^{1g}		\$ 120 B			e a see e
mass spectrometry				D4400 00/00		
Direct current plasma				D4190-82(88)	**.	Note 36

Parameter, Units & Methods	EPA ¹	SW-846 ^{11,7}	Standard Methods ^{2,2m}	ASTM ³	USGS ⁴	Other
21. Color, Platinum Cobalt units or dominant wavelength hue,					ti v v v v	
luminance, purity:		•				
Colorimetric, ADMI	110.1		2120 E			Note 20
	110.1		2120 B	14+	I-1250-85	11010 20
Spectrophotometric	110.3		2120 C		1 1200 00	
	110.5			·		
22. Copper, mg/L: Digestion ⁶ followed by:		*		•		1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
AA direct aspiration ^{6m} ,	220.1	7201	3111 B or C	D1688-90(AorB)		974.27 ⁵
AA furnace,	220.2 or	7211	3113 B	D1688-90(C)	I-3270-85	
	200,91g		*	,	15.15	1
Inductively coupled plasma6m	200.7^{1g}	6010A	3120 B		$(x_{i+1},\dots,x_{i+1},x_{i+1}) \in \mathbb{R}^d$	
Inductively coupled plasma- mass spectrometry	200.8 ^{1g}	6020		• •		
Direct current plasma6m,				D4190-82(88)	21 - 4	Note 36
Colorimetric (Neocuproine), or			3500-Cu D or E			
Bicinchoninate	:					Note 21
23. Cyanide – Total, ug/L:		, 3			-	**
Manual distillation with MgCl ₂			4500-CN-C		14	
Followed by: titrimetric,			4500-CN-D			
Manual or	335.2	9010A	4500-CN-E	D2036-91(A)	I-3300-85	
Automated ²²	335.3	9010A		*. '	. Tale	-
spectrophotometric, or	00 a 41m	0010	•		•	
Semi-automated colorimetry	335.4 ^{1m}	9012				
24. Cyanide amenable to chlorina-	335.1		4500-CN-G	D2036-91(B)	· ·	
tion,					• " "	
ug/L: Manual distillation with						
MgC ₁₂ followed by titrimetric, manual or automated						
spectrophotometric		9010A		4		
		9010A				
25. Fluoride – Total, mg/L:				9		** *
Manual distillation ⁸			4500-F-B	D1170 00/D)	3.00	
Pollowed by manual or	340.2		4500-F-C	D1179-88(B)	T 4007 05	
automated electrode,	240.1		4600 E D	D1170 00(A)	I-4327-85	
SPADNS, Ion chromatography,	340.1 300.0 ^{1m}	0056	4500-F-D	D1179-88(A)		
Or automated complexone	340.3	9030	4500-F-E	1 5	·	2.4
-		-		1.5 %		
26. Gold, mg/L: Digestion ⁶ followed by:			:			i
AA direct aspiration	231.1	. 1	3111 B	Cylia		1.50
AA furnace,	231.2		3113 B	200		
Direct current plasma, or				1.29 gr	port of the contract of the co	Note 36
Inductively coupled plasma	200.7 ^{1g}	6010A		e e		+ i
27. Hardness – Total as CaCO ₃ ,						
mg/L:					•	
Automated colorimetric,	130.1					
EDTA titration,	130.2		2340 C	D1126-86(92)	1-1338-85	973.52B ⁵
or the sum of Ca and Mg as						
their respective carbonates (by				5.4.1	:	Fr. e
ICP or AA direct aspiration)			2340 B			•
(See Parameters 13 and 33)					·	
28. Hydrogen ion (pH), pH units:				and the second	1	,
Electrometric Measurements	150.1	9040B	4500-H+ B	D1293-84(90)	I-1586-85	973.41 ⁵
or		•		(A or B)		
Automated Electrode						Note 23

List	List of Approved Inorganic Test Procedures for Wastewater								
Parameter, Units & Methods	EPA ¹	SW-846 ^{11,7}	Standard Methods ^{2,2n1}	ASTM ³	USGS ⁴	Other			
29. Iridium, ug/L:									
Digestion ⁶ followed by:	225 1		2111 D	4	7 4 4 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1				
AA direct aspiration, AA furnace, or	235.1 235.2		3111 B	*	· · · ·				
Inductively coupled plasma	200,7 ^{1g}	6010A	100						
30. Iron, mg/L:					,				
Digestion ⁶ followed by:									
AA direct aspiration ^{6m} ,	236.1	7380	3111 B or C	D1068-90(AorB)	I-3381-84	973.275			
AA furnace,	236.2 or	7381	3113 B	D1068-90(C)		1.00%			
	200.9 ^{1g}				Target (1)				
Inductively coupled plasma ^{6m} ,	200.7 ^{1g}	6010A	3120 B	D4100 00/00\		Note 36			
Direct current plasma ^{6m} , or Colorimetric (Phenanthroline)		•	3500-Fe D	D4190-82(88) D1068-90(D)		Note 24			
			3300-re D	D1009-30(D)		Note 24			
31. Kjeldahl nitrogen – Total (as N), mg/L:					and the second	275			
Digestion and distillation	351.3		4500-NorgBorC	D3590-89(A)					
Followed by titration	351.3	1 27	4500-NH ₃ E	D3590-89(A)		937.46 ⁵			
Nesslerization or	351.3	• •	4500-NH ₃ C	D3590-89(A)	18 0 0 5 5	201710			
Electrode,	351.3		4500-NH ₃ ForG	20070 07(11)					
Automated phenate,	351.1		4500-NH ₃ H	19	I-4551-788	: .			
Semi-automated block digester,	351.2 ^{1m}		,	D3590-89(B)		and the second			
Or potentiometric	351.4			D3590-89(A)					
32. Lead, mg/L:				****		and the first			
Digestion ⁶ followed by:						:			
AA direct aspiration ^{6m} ,	239.1	7420	3111 B or C	D3559-90(AorB)	I-3399-90	974.27 ⁵			
AA furnace,	239.2 or	7421	3113 B	D3559-90(C)		the state of			
	200.9 ^{1g}	1.00	1.1.1		. 1				
Inductively coupled plasma ^{6m} ,	200.7 ^{1g}	6010A	3120 B	to the second second		1			
Inductively coupled plasma-	200.8^{1g}	6020							
mass spectrometry Direct current plasma ^{6m} ,		W 42		D419082(88)		Note 36			
Voltametry ¹³ or				D3559-90(C)		11016 30			
Colorimetric (Dithizone)			3500-Pb D	25 7 50(0)					
33. Magnesium, mg/L:				···		· . /			
Digestion ⁶ followed by:									
Atomic absorption,	242.1	7450	3111 B	D511–92(B)	I-3447-85	974.27 ⁵			
Inductively coupled plasma,	200.7 ^{1g}	6010A	3120 B						
Direct current plasma, or			4500 15 D			Note 36			
Gravimetric		·	3500-Mg D			:			
34. Manganese, mg/L: Digestion ⁶ followed by:									
AA direct aspiration ^{6m} ,	243.1	7460	3111 B	D858-90 (AorB)	I-3454-85	974.27 ⁵			
AA furnace,	243.1 or		3113 B	D858-90(C)	1-3434-03	914.21			
AA Tuttique,	200.9 ^{1g}	7401	3113 B	D030-90(C)					
Inductively coupled plasma6m,	200.71g	6010A	3120 B			4			
Inductively coupled plasma-	200.8 ^{1g}	6020	5120 B						
mass spectrometry,									
Direct current plasma6m,				D4190-82(88)	*	Note 36			
Colorimetric (Persulfate), or			3500-Mn D			920.205^3			
Periodate		·				Note 25			
35. Mercury – Total ⁶ , ug/L:	_								
Cold vapor AA, manual or	245.1 ^{1g}	7470A	3112 B	D3223-91	I-3462-85	977.22 ⁵			
automated, or	245.2	<u> </u>							
35m. Mercury - Hg(II) and									
organomercurials, ug/L:									
HPLC with electrochemical	245.3 ^{1g}					73.7			
detection	,								

Parameter, Units & Methods	EPA ¹	SW-846 ^{11,7}	Standard Methods ^{2,2m}	ASTM ³	USGS ⁴	Other
36. Molybdenum, mg/L: Digestion ⁶ followed by: AA direct aspiration, AA furnace, Inductively coupled plasma,	246.1 246.2 200.7 ^{1g}	7480 7481 6010A	3111 D 3113 B 3120 B		I3490-85	. : . :
Inductively coupled plasma- mass spectrometry, or Direct current plasma	200.8 ^{1g}	6020			*	Note 36
37, Nickel, mg/L:	4 - 11 -					
Digestion ⁶ followed by: AA direct aspiration ^{6m} , AA furnace,	249.1 249.2 or 200.9 ^{1g}	7520	3111 B or C 3113 B	D1886-90(AorB) D1886-90(C)	I-3499-85	* * * * * * * * * * * * * * * * * * * *
Inductively coupled plasma ^{6m} , Inductively coupled plasma—	200.9 ^{1g} 200.8 ^{1g}	6010A 6020	3120 B			
mass spectrometry, Direct current plasma ^{6m} , or Colorimetric (Heptoxime)		51 55 - QV 550	3500-Ni D	D4190-82(88)	221 - 1 	Note 36
38. Nitrate (as N), mg/L: Brucine sulfate, or Nitrate-nitrite N minus Nitrite N	352.1	in the second se	·		•	973.50 ⁵ ,419D ¹⁹
(see parameters 39 and 40) Ion chromatography	300.0 ^{1m}	9056				
39. Nitrate-nitrite (as N), mg/L: Cadmium reduction, manual or automated, or automated hydrazine Ion chromatography	353.3 353.2 ^{1m} 353.1 300.0 ^{1m}	9056	4500–NO ₃ E 4500–NO ₃ F 4500–NO ₃ H	D3867-90(B) D3867-90(A)	I–4545–85	
40. Nitrite (as N), mg/L: Spectrophotometric, manual or automated (Diazotization), or Ion chromatography ³⁹	354.1 300.0 ^{1m}	9056	4500-NO ₂ B		I-4540-85	Note 27
41. Oil and grease—Total recoverable, mg/L: Gravimetric (freon extraction) Gravimetric (hexane extraction)	413.1 1664	9070 :	5520 B		 154-14	Constitution of the second sec
42. Organic carbon – Total (TOC), mg/L: Combustion or oxidation, Persulfate oxidation	415.1 415.21 ^m	9060	5310 B or D 5310C	D2579-85 (AorB)		973.47 ⁵ p.142 ⁶
43. Organic nitrogen (as N), mg/L: Total Kjeldahl N (Parameter 31) minus animonia N (Parameter 4)						
44. Orthophosphate (as P), mg/L: Ascorbic acid method, automated	365.1	٠.	4500-P F		I-4601-85	1
Or manual single reagent or Manual two reagent, or Ion chromatography	365.2 365.3 300.0 ^{1m}	9056	4500–P E	D515-88(A)		973.55 ⁵
45. Osmium, ug/L: Digestion ⁶ followed by: AA direct aspiration, AA furnace, or Inductively coupled plasma	252.1 252.2 200.7 ^{1g}	7550 6010A	3111 D			-

List o	f Approv	ed Inorganic	Test Procedure	es for Wastewater	-	
Parameter, Units & Methods	EPA ¹	SW-846 ^{11,7}	Standard Methods ^{2,2m}	ASTM ³	USGS ⁴	Other
46. Oxygen, dissolved, mg/L: Winkler (Azide modification) Or electrode	360.2 360.1		4500-O C 4500-O G	D888-92(A) D888-92(B)	I15757810 I15767810	973.45B ⁵
47. Palladium, mg/L: Digestion ⁶ followed by: AA direct aspiration, AA furnace, Direct current plasma, or Inductively coupled plasma	253.1 253.2 200.7 ^{1g}	6010A	3111 B		: · · · · · · · · · · · · · · · · · · ·	Note 36
48. Phenols, ug/L: Manual distillation ²⁸ Followed by manual Or automated ²² colorimetric (4AAP), or Semi-automated colorimetric	420.1 420.1 420.2 420.4 ^{1m}	9065 9066	5530 B 5530 D			Note 29
49. Phosphorus (elemental), mg/L: Gas-Liquid chromatography						Note 30
50. Phosphorus - Total, mg/L: Persulfate digestion Followed by manual or	365.2 365.2 or		4500-P B,5 4500-P E	DE1E 00 (A)		973.55 ⁵
Automated ascorbic acid Reduction, or semi-automated block digestor	365,3 365,1 ^{1m} 365,4		4500-P F	D515-88 (A)	I-4600-85	973.56 ⁵
51. Platinum, mg/L: Digestion ⁶ followed by: AA direct aspiration, AA furnace, Direct current plasma, or Inductively coupled plasma	255.1 255.2 200.7 ^{1g}	6010A	3111 B			Note 36
52. Potassium, mg/L: Digestion ⁶ followed by: Atomic absorption, Inductively coupled plasma, Flame photometric, or Colorimetric (cobalt nitrate)	258.1 200.7 ^{1g}	7610 6010A	3111 B 3120 B 3500–K D		I-3620-85	973.53 ⁵ 317B ¹⁹
53. Residue – total, (total solids), mg/L: Gravimetric 103–105°C	160.3		2540 B		I-3750-85	447
54. Residue – filterable, (TDS), mg/L: Gravimetric, 180°C	160.1		2540 C	:	I-1750-85	14 (4) (4) (4) (4) (4) (4) (4) (4
55. Residue – nonfilterable, (TSS), mg/L: Gravimetric, 103–105°C post washing of residue	160.2		2540 D		I-3765-85	i de la composition della comp
56. Residue – settleable, mg/L: Volumetric (Imhoff cone) or gravimetric	160.5	,	2540 F	-	٠.	
57. Residue – volatile mg/L: Gravimetric, 550°C	160.4		2540 E ³⁸		I-3753-85	

List o	f Approv	ed Inorganic	Test Procedures	for Wastewater	r	
Parameter, Units & Methods	EPA ¹	SW-846 ^{11,7}	Standard Methods ^{2,2m}	ASTM ³	- USGS ⁴	Other
58. Rhodium, ug/L: Digestion ⁶ followed by: AA direct aspiration, AA furnace, or Inductively coupled plasma	265.1 265.2 200.7 ^{1g}	6010A	3111 B		i i i i i i i i i i i i i i i i i i i	
59. Ruthenium, ug/L: Digestion ⁶ followed by: AA direct aspiration, AA furnace, or Inductively coupled plasma	267.1 267.2 200.7 ¹ 8	601 0 A	3111 B			
60. Selenium, ug/L: Digestion ⁶ followed by: AA furnace,	270.2 or	7740	3113 B	E P P P P P P P P P P P P P P P P P P P		tot og skalende for De forske blever De forst
Inductively coupled plasma ^{6m} , Inductively coupled plasma	200,9 ^{1g} 200,7 ^{1g} 200,8 ^{1g}	6010A 6020	3120 B		a sa kanala Kanala ka sa ka	A START SERVE OF TORRES
mass spectrometry, or AA (gaseous hydride)		7741A	3114 B ³⁷	D3859-88(A)	I-3667-85	
61. Silica – Dissolved, mg/L: 0.45 micron filtration: Followed by manual or automated colorimetric	370.1		4500-Si D	D859-88	I-1700-85 I-2700-85	
(Molybdosilicate), or Inductively coupled plasma ⁶	200.7 ^{1g}	6010A	3120 B			
62. Silver ³¹ , mg/L: Digestion ⁶ followed by: AA direct aspiration, AA furnace, Colorimetric (Dithizone), Inductively coupled plasma, Inductively coupled plasma—	200.9 ^{1g} 200.7 ^{1g} 200.8 ^{1g}	7760A 7761 6010A 6020	3111 B or C 3113 B 3120 B		I-3720-85	973.27 ⁵ 319B ¹⁹
mass spectrometry, Or direct current plasma			,			Note 36
63. Sodium, mg/L: Digestion ⁶ followed by: Atomic absorption, Inductively coupled plasma, Direct current plasma, or Flame photometric	273.1 200.7 ^{ig}	7770 6010A	3111 B 3120 B 3500–Na D	D1428-82(A)	I-3735-85	973.54 ⁵ Note 36
64. Specific conductance, micromhos/cm at 25°C: Wheatstone bridge	120.1	9050	2510 B	D1125-91(A)	I1780-85	973.40 ⁵
65. Sulfate (as SO ₄), mg/L: Automated colorimetric (barium chloroanilate),	375.1	9035			* *;	
Semi–automated colorimetric (methylthymol blue) Gravimetric, Turbidimetric, or Ion chromatography	375.2 ^{1m} 375.3 375.4 300.0 ^{1m}	9038	4500–SO ₄ ² CorD	D516-90	14 1 *	925.54 ⁵ 426C ³²
66. Sulfide (as S), mg/L: Titrimetric (iodine) or Colorimetric (methylene blue)	376.1 376.2	•	4500-S ² -E 4500-S ² -D		I-3840-85	228A ³³
67. Sulfite (as SO ₃), mg/L: Titrimetric (iodine-iodate)	377.1		4500-S0 ₃ ²⁻			
68. Surfactants, mg/L: Colorimetric (methylene blue)	425.1		5540 C	D2330-88		

List o	ı Approv	ea Inorganic		es for Wastewater	•	•
Parameter, Units & Methods	EPA ¹	SW-846 ^{11,7}	Standard Methods ^{2,2m}	ASTM ³	USGS ⁴	Other
69. Temperature, °C: Thermomet-	170.1		2550 B			Note 34
ric					and the second	8.4
70. Thallium, ug/L:			de la			,
Digestion ⁶ followed by:						
AA direct aspiration,	279.1	7840	3111 B			
AA furnace.	279.2 or	7841	3113 B			
and the second second	200.91g				4	
Inductively coupled plasma, or	200.7^{1g}	6010A			* * * * * * * * * * * * * * * * * * *	
Inductively coupled plasma-	200.81g	6020		$S_{i,j} = \{ (i,j) \mid i \in \mathcal{I}_{i,j} = 1 \}$		
mass spectrometry		1 (•		in the second
71, Tin, ug/L;				. •	AL ALLES	:
Digestion ⁶ followed by:	v	114				en e
AA direct aspiration,	282.1	7870	3111 B	and the second second	I-3850-7810	•
AA furnace, or	282.2 or		3113 B	•		
THE Identity of	200.9 ^{1g}				· · · · · · · · · · · · · · · · · · ·	
Inductively coupled plasma	200.718	6010A			to the second	
72. Titanium, mg/L:						
Digestion ⁶ followed by:			**	r	and the second of	
AA direct aspiration,	283.1		3111 D			
AA furnace,	283.2		3113 B			
Direct current plasma, or	-0012	. •			•	Note 36
Inductively coupled plasma	200.71g	6010A				
73. Turbidity, NTU: Nephelometric	180.1 ^{1m}	· · · · · · · · · · · · · · · · · · ·	2130 B	D1889-88(A)	I-3860-85	
74. Vanadium, mg/L:						
Digestion ⁶ followed by:			•			
AA direct aspiration,	286.1	7910	3111 D			
AA furnace,	286.2	7911	3113 B	1 1 1		•
Inductively coupled plasma,	200.7 ^{1g}	6010A	3120 B			
Inductively coupled plasma-	200.8^{1g}					;
mass spectrometry			g de la		ALC: NO.	
Direct current plasma, or				D4190-82(88)	* ************************************	Note 36
Colorimetric (Gallic acid)			3500-V D			
75. Zinc, mg/L:						,
Digestion ⁶ followed by:		Ü,			an egypta i film	
AA direct aspiration ^{6m} ,	289.1	7950	3111 B or C		I-3900-85	974.27 ⁵
AA furnace,	289.2 or	7951	3113 B	4.0	Market St.	*
	200.9 ^{1g}					
Inductively coupled plasma6m,	200.7 ^{1g}	6010A	3120 B			
Inductively coupled plasma-	200,8 ^{1g}	6020		*		
mass spectrometry,	#1			4	***	
Direct current plasma6m,	:			D4190-82(88)		Note 36
Colorimetric (Dithizone), or	•		3500-Zn E			
Colorimetric (Zincon)			3500-Zn F			Note 36

¹ "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, United States Environmental Protection Agency, Revised March 1983 and 1979 where applicable. Available from National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161 (703) 487-4650.

^{1g} "Methods for the Determination of Metals in Environmental Samples", EPA-600/4-91-010, Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268, June 1991. Available from the National Technical Information Service (NTIS), order number PB91-231498, 5258 Port Royal Road, Springfield, Virginia 22161, (703) 487-4650.

^{1m} "Methods for the Determination of Inorganic Substances in Environmental Samples", EPA-600/R-93-100, Environmental Protection Agency, August 1993, Office of Research and Development, Washington D.C. 20460, August 1993. Available from NTIS, 5285 Port Royal Road, Springfield, Virginia 22161 (703) 487-4650.

² "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 18th Edition, 1992. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

²m The 18th edition of "Standard Methods for the Examination of Water and Wastewater" is not significantly different from the 17th edition. The 17th edition remains an acceptable reference for those methods which cite the 18th edition.

- 3 "1993 Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1993. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.
- 4 "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments", U.S. Department of the Interior, U.S. Geological Survey, Open-File Report 85-495, 1989, unless otherwise stated. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.
- ⁵ "Official Methods of Analysis of the Association of Official Analytical Chemists", methods manual, 15th Edition (1990). Available from The Association of Official Analytical Chemists, 1111 N. 19th Street, Suite 210, Arlington, VA 22209.
- ⁶ A digestion procedure is required to solubilize suspended material and to destroy possible organic metal complexes. The required digestion procedure(s) for a particular metals analysis is listed in Table BM, Metals Digestion Procedures. Use of the graphite furnace AA technique, inductively coupled plasma, direct current plasma, as well as determination for certain elements such as arsenic, mercury, selenium, silver, and titanium require a modified digestion procedure. In all cases, the analytical method should be consulted for specific instructions and cautions.

If a digestion procedure is given in the determinative method for any of the metals in table B, and this digestion is not listed in table BM, the procedure given in the analytical method should be used however if the digestion included in one of the approved non-EPA references (e.g. "Standard Methods for the Examination of Water and Wastewater") is significantly different from one of the EPA procedures listed in table BM, than the EPA procedure from table BM should be used.

Sample digestion may be omitted for AA (direct aspiration or graphite furnace), direct current plasma, and inductively coupled plasma analyses provided the sample solution to be analyzed meets the following criteria:
(a) has a low COD (<20),

(b) is visibly transparent with a turbidity measurement of 1 NTU or less,

(c) is colorless with no perceptible odor, and

- (d) is of one liquid phase and free of particulate or suspended matter following acidification.
- ^{6m} Either of the following microwave digestion procedures may be used:
- "Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals", CBM corporation, P.O. Box 200, Mattews, North Carolina 28106–0200, April 16, 1992. Available form the CBM Corporation.
 "Test Methods for Evaluating Solid Waste", SW-846 method 3015. United States EPA SW-846, 3rd Edition. Footnote 11 lists the

complete reference.

- ⁷ SW-846 series 6000 and 7000 methods include SW-846 method 7000A, the general AA method description.
- ⁸ Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.
- Ammonia, Automated Electrode Method, Industrial Method Number 379-75WE, dated February 19, 1976, Technicon AutoAnalyzerII. Available from Technicon Industrial Systems, Benedict Avenue, Tarrytown, NY 10591.
- 10 The approved method is that cited in "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments", USGS TWRI, Book 5, Chapter A1 (1979). Available on inter-library loan.
- 11 "Test Methods for Evaluating Solid Waste", 3rd Edition, SW-846, Office of Solid Waste and Emergency Response, Environmental Protection Agency, November 1986, including July 1992, August 1993, September 1994 and January 1995 updates, Washington D.C. 20460. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington DC, (202) 512-1800.
- 12 "Selected Analytical Methods Approved and cited by the United States Environmental Protection Agency", Supplement to the Fifteenth Edition of "Standard Methods for the Examination of Water and Wastewater," from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005, 1981. Available on inter-library loan.
- 13 The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.
- ¹⁴ Carbonaceous biochemical oxygen demand (CBOD₅) must not be confused with the traditional BOD₅ test which measures "total BOD₅." The addition of the nitrification inhibitor is not a procedural option, but must be included to report the CBOD₅ parameter. A discharger whose permit requires reporting the traditional BOD₅ may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states CBOD₅ is required, can the permittee report data obtained using the nitrification inhibitor.
- 15 OIC Chemical Oxygen Demand Method. Available from Oceanography International Corporation, 512 West loop, P.O. Box 2980, College Station, TX 77840.
- ¹⁶Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.
- ¹⁷ The back titration method will be used.
- ¹⁸ ORION Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977. Available from Orion Research Incorporated, 840 Memorial Drive, Cambridge, MA 02138.
- ¹⁹ The approved method is that cited in the "Standard Methods for the Examination of Water and Wastewater", 14th Edition, 1976. Available on inter-library loan.
- 20 "An Investigation of Improved Procedures for Measurement of Mill Effluent and Receiving Water Color", NCASI Technical Bulletin No. 253. December, 1971. Available from National Council of the Paper Industry for Air and Stream Improvements, Inc., 260 Madison Avenue, New York, NY 10016.
- ²¹ Copper, Bicinchoninate Method, Method 8506, Hach Handbook of Water Analysis, 1979. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

- ²² After the manual distillation is completed, the auto-analyzer manifolds in EPA Methods 335.03 (Cyanide) or 420.2 (phenols) are simplified by connecting the re-sample line directly to the sampler. When using the manifold setup shown in Method 335.3, the buffer 6.2 should be replaced with the buffer 7.6 found in Method 335.2.
- Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976, Technicon AutoAnalyzer II. Available from Technicon Industrial Systems, Benedict Avenue, Tarrytown, NY 10591.
- ²⁴ 1, 10-Phenanthroline Method for Iron, Hach Method 8008, 1980. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.
- ²⁵ Periodate Oxidation Method for Manganese, Method 8034. Hach Handbook of Wastewater Analysis, 1979, pp. 2–113 and 2–117. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.
- 26 "Methods for Analysis of Organic Substances in Water", by D. F. Goerlitz and Eugene Brown: USGS—TWRI, Book 5, Chapter A3, p. 4, 1972. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.
- ²⁷ Nitrite Nitrogen, Hach Method 8507. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.
- ²⁸ Just prior to distillation, adjust the sulfuric acid preserved sample to pH 4 with 1 + 9 NaOH.
- ²⁹ The approved method is that cited in "Standard Methods for the Examination of Water and Wastewater", 14th Edition. The colorimetric reaction is conducted at a pH of 10.0 + 0.2. The approved methods are given on pp. 576-81 of the 14th Edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectrophotometric procedure. Available on inter-library loan.
- 30 "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography", by R. F. Addison and R. G. Ackman, Journal of Chromatography, Volume 47, No. 3, pp. 421-426, 1970. Available in most public libraries. Back volumes of the Journal of Chromatography are available from Elsevier/North-Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164
- ³¹ Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to a pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2M Na₂S₂O₃ and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.
- 32 The approved method is that cited in "Standard Methods for the Examination of Water and Wastewater", 15th Edition. Available on inter-library loan.
- 33 The approved method is that cited in "Standard Methods for the Examination of Water and Wastewater", 13th Edition. Available on inter-library loan.
- 34 "Water Temperature-Influential Factors, Field Measurement, and Data Presentation", by H. H. Stevens, Jr., J. Ficke, and G. F. Smoot: USGS-TWRI Book 1, Chapter D1, 1975. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.
- 35 Zincon Method of Zinc Method 8009. Hach Handbook for Water Analysis, 1979, pp. 2-231 and 2-333. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.
- ³⁶ Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method AES0029, "1986 Revised 1991, Fison Instruments, Inc., 32 32 Commerce Center, Cherry Hill Drive, Danvers MA 01923.
- ³⁷ Use the digestion given in the method.
- ³⁸ The temperature must be maintained between 500–550° C, and not the temperature listed in the method.
- ³⁹ Nitrate-nitrite determinations by ion chromatography must be analyzed within 48 hours.

Table BM Metals Digestion Procedures

Analysis	SW-846 ¹	EPA ²	EPA ³
Dissolved Metals ⁴	3005A,3040A ¹⁰		4,1.1
Suspended Metals ⁵	3005A		4.1.2
Total Metals ⁶	3010A, 3020A ¹¹ , 3050A ¹⁰ , 3051A ¹⁰		4.1.3
Total Recoverable Metals ⁷	3005A	200.2	4.1.4
Acid Soluble Metals ⁸		200.112	
Available Metals ⁹	3015 ¹³		

¹ "Test Methods for Evaluating Solid Waste", 3rd Edition, SW-846, Office of Solid Waste and Emergency Response, Environmental Protection Agency, November 1986, including December 1987, July 1992, August 1993, September 1994 and January 1995 updates, Washington D.C. 20460. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington DC 20402, (202) 512–1800.

^{2&}quot;Methods for the Determination of Metals in Environmental Samples", EPA-600/4-91-010, Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268, June 1991. Available from the National Technical Information Service (NTIS), order number PB91-231498, 5258 Port Royal Road, Springfield, Virginia 22161, (703) 487-4650.

- 3"Methods for Chemical Analysis of water and Wastes", EPA-600/4-79-020, United States Environmental Protection Agency, Revised March 1983 and 1979 where applicable, Available from National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161 (703) 487-4650.
- 4"Dissolved metals" means those constituents of a sample that will pass through a 0.45 micron membrane filter prior to sample acidification.
- 5"Suspended metals" means the concentration of metals determined in the portion of a sample retained by a 0.45 micron membrane filter prior to acidification.
- 6"Total metals" means the concentration of metals determined on a solid sample or unfiltered aqueous sample following a vigorous digestion, or alternatively the sum of the metals determined in both the dissolved and suspended fractions.
- 7"Total recoverable metals" means the concentration of metals determined on an unfiltered sample following treatment with hot dilute mineral acid.
- 8"Acid soluble metals" means those constituents of a sample that will pass through a 0.45 micron membrane filter after the sample has been adjusted to pH 1.75 and held for 16 hours. This method is applicable to arsenic, cadmium, chromium, copper, and lead.
- 9"Available metals" are equivalent to "total metals". SW-846 lists method 3015 as a preparation for available metals.
- ¹⁰ These methods are for total metals analysis of sediment, sludge, and soil samples and do not apply to wastewater. The required analytical methodology for metals in wastewater sludge is given in Table EM.
- ¹¹Method 3020 is applicable for analysis by GFAA. Method 3010 requires sample acidification with HCl.
- ¹²Method 200.1 is only applicable for As, Cd, Cr, Cu and Pb.
- ¹³This method is a microwave-assisted acid leachate digestion.

Table C
List of Approved Test Procedures for Non-Pesticide Organic Compounds in Wastewater

	•	EPA Method					thod Numbe	r11,12		
	and the state of t		mber ^{1,6}	Standard	GC	GC	GC/MS	GC/MS		
	Parameter	GC	GC/MS	Methods ^{8,13}	capillary	pkd ¹⁴	capillary	pkd ¹⁴	Other	
•	Volatiles		624 ³		8021A		8260A	8240B		
	A. Halogenated volatiles	601	1624	6230 B, 6210 B	·	. 8010B		· -		
	Bromodichloromethane			1 - 4				1		
	Bromoform									
	Bromomethane	. :		1,141			1.5			
	Carbon tetrachloride		\$	4					Note 2, p.130	
	Chloroethane									
	Chloroform			100			1.00		Note 2, p.130	
	Chloromethane									
	Dibromochloromethane	1, 1	et ***	the second						
	Dichlorodifluoromethane	100	· · · · · · · · · · · · · · · · · · ·	not 6210 B				* / * *	***	
	1,1-Dichloroethane	•					."			
	1,2-Dichloroethane									
	1,1-Dichloroethene									
	trans1,2-Dichloroethene									
	1,2-Dichloropropane									
	cis-1,3-Dichloropropene	i.								
	trans-1,3-Dichloropropene								•	
	Methylene chloride								Note 2, p.130	
	1,1,2,2-Tetrachloroethane					•			Note 2, p.130	
	Tetrachloroethene								Note 2, p.130	
	1,1,1-Trichloroethane						. •			
	1,1,2-Trichloroethane								Note 2, p.130	
	Trichloroethene									
	Trichlorofluoromethane									
	Vinyl chloride									
	B. Aromatic volatiles	602	. ,	6220B		8020A		<u></u>	ž e	
	Benzene	•	1624	6210B				:		
	Chlorobenzene	601	1624	6210B,					Note 2, p.130	
				6230B						
	1,2-Dichlorobenzene	601,	625,	6230B,				**		
		612	1625	6410B						

Table C
List of Approved Test Procedures for Non–Pesticide Organic Compounds in Wastewater

	(Fig. 1)		Method		SW	-846 Met	lhod Numbe	r ^{11,12}	
	1.4	Nu	mber ^{1,6}	Standard	GC	GC -	GC/MS	GC/MS	
	Parameter	···GC	GC/MS	Methods ^{8,13}	capillary	pkd ¹⁴	capillary	pkd ¹⁴	Other
. 1	1,3-Dichlorobenzene	601,	625,	6230B,	-/				:
	1,4-Dichlorobenzene	612 601, 612	1625 625, 1625	6410B 6230B, 6410B		. \$			
	Ethylbenzene		1624	6210B					
	Toluene		1624	6210B					4.8
C.	Other volatiles	603	1624,624		8030A		8260A	8240B	
	Acrolein		•			- .			LC:8315 (SW-846)
	Acrylonitrile				8031			1,4	LC: 8316 (SW-846)
II. Ph	nenols	604	625, 1625	6410B, 6420B		8040 A	8270B	8250A	
4_	-Chloro-3-methylphenol								
2-	-Chlorophenol	•							
2,	4-Dichlorophenol								
2,	4-Dimethlyphenol			•					
	4-Dinitrophenol								
	-Methyl-4,6-dinitrophenol								•
2-	-Nitrophenol								
4-	-Nitrophenol								.,
Pe	entachlorophenol								Note 2, p.14
Pł	henol			•					
2,	4,6-Trichlorophenol								,
III. Pl	hthalate esters	606	625, 1625	6410 B	8061	8060	8270B	8250A	
В	enzyl butyl phthalate		. -						
B	is(2-ethylhexyl)phthalate	1 1 1 2 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1							
D	iethyl phthalate		* : "						•
D	imethyl phthalate							,	
D	i-n-butyl phthalate								
Đ	i-n-octyl phthalate		. 1 3.						J.
IV N	litrosamines	607	625, 1625	6410 B		8070	8270B	8250A	
N!	-Nitrosodimethylamine	7 H	note 4						***
	I-Nitrosodi-n-propylamine		HOIC 4						tijeta i laeta
	I-Nitrosodiphenylamine		note 4						
	olychlorinated biphenyls	608	625	6410 B	8081	8080	8270B	8250A	Note 2, p.43
D	ICD 1016		1			Α			
	CB-1016								
	CB-1221 CB-1232								
	CB-1232 CB-1242								
	CB-1242 CB-1248								
	CB-1248 CB-1254	: "							
	CB-1254 CB-1260						•		
VI. N	Vitroaromatics & cyclic	609	625,	6410 B		8090	8270B	8250A	
ketor			1625						
	4.4-Dinitrotoluene								
	2,6-Dinitrotoluene								
	sophorone		*					٠.	
N	Vitrobenzene								

Table C
List of Approved Test Procedures for Non-Pesticide Organic Compounds in Wastewater

			Method				thod Numbe		
	Parameter Parameter	Nur GC	nber ^{1,6} GC/MS	Standard Methods ^{8,1}		GC pkd ¹⁴	GC/MS capillary	GC/MS pkd ¹⁴	· · · Other
711	Polynuclear aromatic	610/FI	625,	6410 B,		8100	8270B	8250A	Note 9; 610,
	Irocarbons	D	1625	6440 B		0100	:	025011	LC: 8310 (SW-846)
	Acenaphthene								
	Acenaphthylene				* (
	Anthracene								
	Benzo(a)anthracene		7		* *	. *			
	Benzo(a)pyrene								
	Benzo(b)fluoranthene Benzo(g,h,i)perylene								٠
	Benzo(k)fluoranthene								
	Chrysene								
	Dibenzo(a,h)anthracene	45.5			431 0		* •		•
	Fluoranthene				it.				
	Fluorene								4.3
	Ideno (1,2-3-cd)pyrene								
	Naphthalene				· 8021A				
	Phenanthrene								# T
	Pyrene						,		.1 1
III.	Haloethers	611	625,	6410 B		8110	8270B	8250A	
	107-70 -4-1		1625						
	Bis(2-chloroethoxy) methane Bis(2-chloroethyl)ether								
	4-Bromophenylphenyl ether								
	4-Chlorophenylphenyl ether								
	2,2-Oxybis (1-chloropropane)	•			\mathcal{P}_{i}		111		1 1 4 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
	Chlorinated hydrocarbons	612	625,	6410 B	8121	8120A	8270B	8250A,	. **
			1625				8260A	8240A	
	Benzyl chloride					8010B	not 8270B	not 8250A	Note 2, p.130 Note 5, p.\$10
	2-Chloronaphthalene						not 8260A	not 8240A	8410 (SW-846)
	Epichlorohydrin					8010B	not	not	Note 2, p.130
	Dpiomoron, and					:.	8270B	8250A	Note 5, p.S10
	Hexachlorobenzene				8081	41.55	not 8260A	not 8240A	8410 (SW-846)
	Hexachlorobutadiene				8021A		020071	not	8410
	Hexachlorocyclopentadiene		note 4		8081	.*	not	8240A not	(SW-846) 8410
	1,2,4-Trichlorobenzene				8021A		8260A	8240A not	(SW-846) Note 2, p.130
								8240A	
	Hexachloroethane							not 8240A	8410 (SW-846)
	Benzidine		note 4				not 8260A	not 8240A	LC: 605
	3,3-Dichlorobenzidine						not 8260A	not 8240A	
ζ.	Polychlorinated dibenzo-p-dic	xins and	1613 A ⁷		ι ——		8280,		
	furans 1,2,3,4,6,7,8-Heptachlorodiber	nzo-p-dioxi	in			• :	8290	-	
							-		
	1,2,3,4,6,7,8-Heptachlorodiber	zofuran		_					

Table C
List of Approved Test Procedures for Non-Pesticide Organic Compounds in Wastewater

	EPA Method		SW-	846 Met	thod Number 11,12	
	Number ^{1,6}	Standard	GC	GC	GC/MS GC/MS	
Parameter	GC GC/M	IS Methods ^{8,13}	capillary	pkd ¹⁴	capillary pkd ¹⁴	Other
1,2,3,4,7,8-Hexachlorodibenzo-p -	-dioxin					
1,2,3,6,7,8-Hexachlorodibenzo-p-c	dioxin					
1,2,3,7,8,9-Hexachlorodibenzo-p-c	lioxin					
1,2,3,4,7,8-Hexachlorodibenzofura	n ·					
1,2,3,6,7,8-Hexachlorodibenzofurar	n				and the second second	
1,2,3,7,8,9-Hexachlorodibenzofurat	n					
2,3,4,6,7,8-Hexachlorodibenzofura	n					
Octachlorodibenzo-p-dioxin		-				
Octachlorodibenzofuran						
1,2,3,7,8-Pentachlorodibenzo-p-di	oxin	200	ti .		4.	
1,2,3,7,8-Pentachlorodibenzofuran	•		i i i		1.50	
2,3,4,7,8-Tetrachlorodibenzo-p-die	oxin	211 h	Serie (1940au)		j	
2,3,7,8-Tetrachlorodibenzo-p-diox	in 613 ⁵ m		r 1,		e de la	Note 10
2,3,7,8-Tetrachlorodibenzofuran			**		*4.*	

- ¹"The full text of Methods 601-613, 624, 625, 1624, and 1625, are given in Appendix A of 40 CFR part 136," Test Procedures for Analysis of Organic Pollutants". The standardized test procedure to be used to determine the method detection limit (MDL) for these procedures is given in Appendix B of 40 CFR part 136, "Definition and Procedure for the Determination of the Method Detection Limit." Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.
- 2"Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," Environmental Monitoring and Support Laboratory, United States Environmental Protection Agency, Cincinnati, Ohio 1978. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, 26 W. St. Claire, Cincinnati, Ohio 45268.
- ³Method 624 may be extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, the preferred method for these two compounds is Method 603 or Method 1624.
- ⁴Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625, are preferred methods for these compounds.
- 5"Selected Analytical Methods approved and Cited by the United States Environmental Protection Agency," Supplement to the 15th Edition of "Standard Methods for the Examination of Water and Wastewater" (1981). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20036.
- 5m625 Sreening only.
- 6Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 601-613, 624, 625, 1613A, 1624, and 1625 in accordance with procedures in section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going basis must spike and analyze 10% (5% for Methods 624 and 625 and 100% for Methods 1624 and 1625) of all samples to monitor and evaluate laboratory data quality in accordance with sections 8.3 and 8.4 of these Methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance.
- Method 1613 Revision A: Tetra-through Octa- Chlorinated Dioxins and Furans by Isotope Dilution, HRGC/HRMS, Environmental Protection Agency, Federal Register, page 5098, February 1991. Available from the Superintendent of Documents, US Government Printing Office, Washington, D.C. 20402.
- 8"Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 18th Edition, 1992. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.
- Method D4657-92, "Annual Book of Standards- Water and Environmental Technology", Section 11, Parts 11.01 and 11.02, American Society for Testing and Materials, 1993. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.
- 10Method D4675-92, "Annual Book of Standards- Water and Environmental Technology", Section 11, Parts 11.01 and 11.02, American Society for Testing and Materials, 1993. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.
- 11 "Test Methods for Evaluating Solid Waste", 3rd Edition. SW-846, Office of Solid Waste and Emergency Response, Environmental Protection Agency, November 1986, including December 1987, July 1992, August 1993, September 1994 and January 1995 updates, Washington DC 20460. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402, (202) 512-1800.
- ¹²SW-846 methods 8021, 8061, 8081, and 8121 require one of the following sample preparation (extraction/clean-up) procedures: 3500/3510 (liquid-liquid extraction), 3500/3520 (continuous liquid-liquid extraction), or 5030 (purge and trap method). The required sample preparation procedure is given in the determinative procedure. Method 8021 requires 5030 (purge and trap). Methods 8081 and 8121 require either 3500/3510 or 3500/3520 in addition to 3600. Method 8061 requires 3510. For methods 8021, 8061, 8081, and 8121 see also SW-846 method 8000A.
- 13The 18th edition of "Standard Methods for the Examination of Water and Wastewater" is not significantly different from the 17th edition. The 17th edition remains an acceptable reference for those methods which cite the 18th edition.
- ¹⁴In order to reference these methods, the laboratory must use a packed column for the GC separations.

Table D

List of Approved Test Procedures for Pesticides¹ in Wastewater

	:			SW-8		Standard		
	Parameter	Method	EPA ^{2,7}	pkd ¹¹ -	cap.	Methods ^{R,9}	ASTM ^c	Other
l.:	Aldrin	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630B&C 6410B	D3086-90	Note 3, p. 7; Note 4, p. 30
2.	Ametryn	GC						Note 3, p. 83; Note 6, p. 868
	Aminocarb	HPLC				*		Note 10
	Atraton	GC						Note 3, p.83; Note 6, p.S68
i.	Atrazine	GC		8140	8141A			Note 3, p.83; Note 6, p.868
5.	Azinphos methyl	GC GC/MS		8140 8250A	8141A 8270B			Note 3. p.25; Note 6, p.851
7.	Barban	HPLC GC/MS		8250A	8270B			Note 10
3.	α-ВНС	GC GC/MS	608 625 ⁵	8080A 8250A	8081 8270B	6630 B & C 6410 B	D308690	Note 3, p.7
).	β-ВНС	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 C 6410 B	D3086-90	3
10.	δ-ВНС	GC GC/MS	608 625 ⁵	8080A 8250A	8081 8270B	6630C 6410B	D3086-90	
11.	γ-BHC(Lindane)	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630B & C 6410B	D3086-90	Note 3, p. 7; Note 4, p. 30
12.	Captan	GC GC/MS		8250A	8270B	6630B	D3086-90	Note 3, p. 7.
3.	Carbaryl	HPLC GC/MS	+ 13 34	8250A	8270B			Note 10
14.	Carbophenothion	GC GC/MS		8140 8250A	8141A 8270B	,	4 <u>1</u>	Note 4, p.30; Note 6, p.S73
15.	Chlordane	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D3086-90	Note 3, p.7
16.	Chloropropham	HPLC			14			Note 10
7.	2,4-D	GC		8150B	8151	6640 B		Note 3, p.115; Note 4, p.35
18.	4,4'-DDD	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D3086-90	Note 3. p.7; Note 4, p.30
19.	4,4'DDE	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D3086-90	Note 3, p.7; Note 4, p.30
20.	4,4'-DDT	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D3086-90	Note 3, p.7; Note 4, p.30
21.	Demeton-O	GC GC/MS		8140 8250A	8141A 8270B			Note 3, p.25; Note 6, p.S51
22.	Demeton-S	GC GC/MS	V	8140 8250A	8141A 8270B	er i julija		Note 3, p.25; Note 6, p.S51
23.	Diazinon	GC		8140	8141			Note 3, p.25; Note 4, p.30; Note 6, p.S51
24.	Dicamba	GC		8150B	8151		, , , , , , , , , , , , , , , , , , ,	Note 3, p.115
25.	Dichlofenthion	GC	8 81	8140	8141			Note 4, p.30; Note 6, p.S73
26.	Dichloran	GC				6630 B & C	D308690	
27.	Dicofol	GC	-					
28.	Dieldrin :	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	•	Note 3, p.7; Note 4, p.30
29.	Dioxathion	GC GC/MS		8140 8250A	8141A 8270B		· .	Note 4, p.30; Note 6, p.S73
30.	Disulfoton	GC GC/MS		8140 8250A	8141A 8270B		4	Note 3, p.25; Note 6, p.S51
31:	Diuron	HPLC					The same of the	Note 10

Table D
List of Approved Test Procedures for Pesticides¹ in Wastewater

	•	List o	f Approv			s for Pesticide	s ¹ in Wastewa	ter
	D		53n i 27		346 ^{A,8}	Standard	A CUIDS EC	0.0
	Parameter	Method	EPA ^{2,7}	pkd ¹¹	cap.	Methods ^{R,9}	ASTM ^c	Other
32,	Endosulfan I	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D308690	Note 3, p.7
33.	Endosulfan II	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D3086-90	Note 3, p.7
34.	Endosulfan sul- fate	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 C 6410 B		
35.	Endrin	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D3086-90	Note 3, p.7; Note 4, p.30
36.	Endrin aldehyde	GC GC/MS	608 625	8080A 8250A	8081 8270B	6410 B	D3086-90	
37.	Ethion	GC GC/MS		8140 8250A	8141A 8270B			Note 4, p.30; Note 6, p.S73
38.	Fenuron	HPLC			11 1 1 1 1			Note 3, p.104; Note 6, p.S64
39.	Fenuron-TCA	HPLC			-73			Note 10
40.	Heptachlor	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D3086-90	Note 3, p.7; Note 4, p.30
41.	Heptachlor epox- ide	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B 6410 B	D308690	Note 3, p.7; Note 4, p.30; Note 6 p.S73
42.	Isodrin	GC GC/MS		8080A 8250A	8081 8270B			Note 4, p.30; Note 6, p.S73
43.	Linuron	HPLC		er e				Note 10
44.	Malathion	GC GC/MS		8140 8250A	8141A 8270B	6630 C	1 4	Note 3, p.25; Note 4, p.30; Note 6, p.S51
45.	Methiocarb	HPLC	<u>.</u>					Note 10
46.	Methoxychlor	GC GC/MS	1	8080A 8250A	8081 8270B	6630 B & C	D3086-90	Note 3, p.7; Note 4, p.30
47.	Mexacarbate	HPLC GC/MS	*	8250A	8270B	1		Note 10
48.	Mirex	GC GC/MS		8080A 8250A		6630 B & C		Note 3, p.7
49.	Monuron	HPLC	7.11.					Note 10
50.	Monuron-TCA	HPLC			7			Note 10
51.	Neburon	HPLC	A may				:-	Note 10
52.	Parathion methyl	GC GC/MS		8140 8250A	8141A 8270B	6630 C		Note 3, p.25; Note 4, p.30
53.	Parathion ethyl	GC GC/MS	# <u>.</u>	8140 8250A	8141A 8270B	6630 C	D308690	Note 3, p.25
54.	PCNB	GC GC/MS		8080A 8250A	8081 8270B	6630 B & C		Note 3, p.7
55.	Perthane	GC	·	8080A	8081		D3086-90	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
56,	Prometon	GC						Note 3, p.83; Note 6, p.S68
57.	Prometryn	GC		-				Note 3, p.83; Note 6, p.S68
57.	Propazine	GC			•			Note 3, p.83; Note 6, p.S68
58.	Propham	HPLC						Note 10
59.	Propoxur	HPLC						Note 10
60.	Secbumeton	HPLC						Note 10
61.	Siduron	HPLC	•.					Note 10
62.	Simazine	GC		8140	8141A		· ·	Note 3, p.83; Note 6, p.S68
63.	Strobane	GC	· ·	8080A	8081	6630 B & C		Note 3, p.7
64.	Swep	HPLC						Note 10
65.	2,4,5-T	GC		8150B	8151	6640 B		Note 3, p.115; Note 4, p.35

Table D

List of Approved Test Procedures for Pesticides in Wastewater

					846 ^{A,8}	Standard	1	
	Parameter	Method	EPA ^{2,7}	pkd ¹¹	cap.	Methods ^{R,9}	ASTM ^c	Other
66.	2,4,5-TP (Sil- vex)	GC		8150B	8151	6640 B		Note 3, p.115
67.	Terbuthylazine	GC					5 5	Note 3, p.83; Note 6, p.S68
68.	Тохарћеле	GC GC/MS	608 625	8080A 8250A	8081 8270B	6630 B & C 6410 B	D308690	Note 3, p.7; Note 4, p.30
70.	Trifluralin	GC GC/MS		8080A 8080A	8081 8270B	6630 B	1.5.1	Note 3, p.7

A"Test Methods for Evaluating Solid Waste", 3rd Edition. SW-846, Office of Solid Waste and Emergency Response, Environmental Protection Agency, November 1986, including December 1987, July 1992, August 1993, September 1994 and January 1995 updates, Washington DC 20460. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402, (202) 512–1800.

Table E List of Approved Radiological Test Procedures For Wastewater

Pa	rameter and Units	Method	EPA ¹	Standard Methods ²	ASTM ³	USGS ⁴
1.	Alph-Total, pCi per liter	Proportional or Scintillation Counter	900.0	7110 B	D194390	pp. 75 and 78 ⁵
2.	Alpha-Counting error, pCi per liter	Proportional or Scintillation Counter	Appendix B	7110 B	D1943-90	p. 79
3.	Beta-Total, pCi per liter	Proportional Counter	900.0	7110 B	D1890-90	pp. 75 and 78 ⁵

B"Standard Methods for the Examination of Water and Wastewater", 18th Edition, Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 1015 Fifteenth Street, N.W., Washington, D.C. 20005, 1992. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

C"Annual Book of Standards – Water and Environmental Technology", Section 11, Parts 11.01 and 11.02, American Society for Testing and Materials, 1993. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

¹Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table D, where entries are listed by chemical name and type.

²The full text of methods 608 and 625 are given in Appendix A of the Federal Register, October 26, 1984 (Part VIII, 40 CFR part 136), "Test Procedure for Analysis of Organic Pollutants". The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given in Appendix B of 40 CFR part 136, "Definition and Procedure for the Determination of the Method Detection Limit". Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

^{3&}quot;Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater". U.S. Environmental Protection Agency, September, 1978. This EPA publication includes thin-layer chromatography (TLC) methods. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, 26 W. St. Claire, Cincinnati, Ohio 45268.

^{4&}quot;Methods for Analysis of Organic Substances in Water", Book 5, Chapter A3, 1987. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

⁵The method may be extended to include a(alpha)-BHC, d(delta)-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.

^{6&}quot;Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of "Standard Methods for Examination of Water and Wastewater" (1981). Available from: American Public Health Association, 1015 15th St., N.W., Washington, D.C. 20005.

⁷Each analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A in 40 CFR part 136) in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

⁸Some of these methods require a preliminary extraction. Methods 8141 A and 8081 require the use of either SW-846 method 3500/3510 or 3500/3520. Methods 8151 and 8270 B include the extraction steps necessary for most compounds. For methods 8081, 8141, and 8151 see also SW-846 method 8000 A and 3600.

⁹The 18th edition of "Standard Methods for the Examination of Water and Wastewater" is not significantly different from the 17th edition. The 17th edition remains an acceptable reference for those methods which cite the 18th edition.

¹⁰HPLC method 623 from "Methods for Nonconventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater", EPA 440/1-83/079- C, United States Environmental Protection Agency. Available from National Technical Information Service, 5258 Port Royal Road, Springfield, Virginia, 22161 (703) 487-4650.

¹¹In order to reference these methods, the laboratory must use a packed column for the GC separations.

Table E List of Approved Radiological Test Procedures For Wastewater

Pa	rameter and Units	Method	EPA ¹	Standard Methods ²	ASTM ³	USGS ⁴
4,	Beta-Counting error, pCi	Proportional Counter	Appendix B	7110 B	D1890-90	p. 79
5,	(a) Radium-Total	Proportional Counter	903.0	7500Ra B	D2460-90	
	(b) 226Ra, pCi per liter	Scintillation Counter	903.1	7500Ra C	D3454-7991	p. 81

¹ "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/-4-80-032, U.S. Environmental Protection Agency, August 1980.

⁵ The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the "total".

	Approved Ana	Table EM llytical Methods For Sludge	
Parameter	Digestion	Method	Method Number
Metals ¹			
Arsenic	3050A	Inductively Coupled Plasma Emission	6010A
Arsenic	7061A	Gaseous Hydride ²	7061A
Arsenic	3050A	Graphite Furnace	7060A
Beryllium	3050A	Inductively Coupled Plasma Emission	6010A
Beryllium	3050A	Flame Atomic Absorption	7090
Beryllium	3050A	Graphite Furnace	7091
Cadmium	3050A	Inductively Coupled Plasma Emission	6010A
Cadmium	3050A	Flame Atomic Absorption	7130
Cadmium	3050A	Graphite Furnace	7131A
Chromium	3050A	Inductively Coupled Plasma Emission	6010A
Chromium	3050A	Flame Atomic Absorption	7190
Chromium	3050A	Graphite Furnace	7191
Copper	3050A	Inductively Coupled Plasma Emission	6010A
Copper	3050A	Flame Atomic Absorption	7210
Lead	3050A	Inductively Coupled Plasma Emission	6010A
Lead	3050A	Flame Atomic Absorption	7420
Lead	3050A	Graphite Furnace ³	7421
Mercury	7471A	Cold Vapor	7471A
Molybdenum	3050A	Inductively Coupled Plasma Emission	6010A
Molybdenum	3050A	Flame Atomic Absorption	7480
Molybdenum	3050A	Graphite Furnace –	7481
Nickel	3050A	Indúctively Coupled Plasma Emission	6010A
Nickel	3050A	Flame Atomic Absorption	7520
Selenium	3050A	Inductively Coupled Plasma Emission	6010A
Selenium	7741A	Gaseous Hydride ²	7741A
Selenium	3050A	Graphite Furnace	7740
Zinc	3050A	Inductively Coupled Plasma Emission	6010A

² "Standard Methods for the Examination of Water and Wastewater", 17th or 18th Edition, Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 1015 Fifteenth Street, N.W., Washington, D.C. 20005, 1989. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

^{3 &}quot;1993 Annual Book of Standards, Water" Section 11.01 and 11.02, Water and Environmental Technology, American Society for Testing and Materials, 1993. Available from American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

^{4 &}quot;Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survey, Open-File Report 76-177 (1976)

	Table EM Approved Analytical Methods For Sludge									
Parameter	Digestion	Method :/	Method Number							
Zinc	3050A	Flame Atomic Absorption	7950							
Biological			# 4							
Enteric viruses	NA	Centrifuge Concentration	D 4994-89 ⁴							
Fecal coliform	NA	Most Probable Number Membrane Filter	9221 E or 9222 D ⁵							
Helminth ova	NA	Density Gradient Flotation	6							
Specific Oxygen Uptake Rate	NA.	Respirometer	2710 B ⁵							
Salmonella			9260 D.1 ⁵							
Physical		. !								
Solids	NA	Gravimetric	2540 G ⁵							
Percent Volatiles Solids Reduction	NA CONTROL OF	Calculation	8							

¹ Test Methods for Evaluating Solid Waste'', SW-846, Office of Solid Waste and Emergency Response, Environmental Protection Agency, November 1986, including December 1987 and July 1992 updates, Washington, DC 20460. Available from the Superintendent of Documents, U.S. Government Printing Office, Room 190, Federal Building, P.O. Box 371954, Pittsburgh, PA 15250-7954, (202) 783-3238.

⁹If an alternative digestion procedure is specified in the analytical method, the digestion in the method shall be used. In all cases, consult the analytical method for special requirements and cautions. SW-846 method 3051 is an acceptable alternate digestion procedure to SW-846 method 3050A.

	Required Containers	s, Preservation	Table F Techniques, and Holding Times for Waste	water
Param	eter No/name	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
TABLE A – Bacterial Tests:				¥
1-5.	Bacteria	P,G	Cool, 4°C, 0.008%, Na ₂ S ₂ O ₃ ⁵	6 hours
6–7.	Enteroviruses	P,G	Cool, 4°C	24 hours
8.	Mutagenicity	G, Teflon- lined cap	Cool, 4°C	7 days
9-12.	Acute & chronic toxicity	P,G	Cool, 4°C	48 hours
TABL	EB-Inorganic Tests:			
1.	Acidity	P,G	Cool, 4°C	14 days
2.	Alkalinity	P,G	Cool, 4°C	14 days
4.	Ammonia	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
9.	Biochemical oxygen demand	P,G	Cool, 4°C	48 hours
11.	Bromide	P,G	None required	28 days
14.	Biochemical oxygen demand, carbonaceous	P,G	Cool, 4°C	48 hours
15.	Chemical oxygen demand	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days

²High levels of chromium, copper, mercury, silver, cobalt, or molybdenum may interfere with the analysis. Consult method 3114, of "Standard Method for the Examination of Water and Wastewater", 17th or 18th edition, for more information.

³Concentrations of lead in municipal sludge may exceed the working range of Graphite Furnace.

^{4&}quot;1993 Annual Book of ASTM Standards, Section 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1993, 1916 Race Street, Philadelphia, PA 19103. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

^{5&}quot;Standard Methods for the Examination of Water and Wastewater", 18th ed., American Public Health Association, 1015 Fifteenth Street NW, Washington D.C. 20005, 1992. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

^{6&}quot;Occurrence of Pathogens in Distribution and Marketing Municipal Sludges", EPA 600/1-87-014, Environmental Protection Agency, 1987. Available from the National Technical Information Service, order #PB 88-154273/AS, 5285 Port Royal Road, Springfield, Virginia 22161, (703) 487-4650.

^{7-&}quot;Determination and Enumeration of Salmoneila and Pseudomonas aeruginosa", Kenner, B.A. and H.A. Clark, J. Water Pollution Control Federation, 46(9):2163–2171, 1994. Available from the Water Environment Federation, 601 Wythe St., Alexandria, VA 22314.

^{8&}quot;Environmental Regulations and Technology – Control of Pathogens and Bextors in Sewage Sludge", EPA-625/R-92/013, Environmental Protection Agency, Cincinnati, OH, 1992. Available from the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, (703) 487-4650.

	Table F Required Containers, Preservation Techniques, and Holding Times for Wastewater					
Parameter No/name Container 1		Preservation ^{2,3}	Maximum holding time ⁴			
16.	Chloride	P.G	None required	28 days		
17.	Chlorine, total residual	P,G	None required	Analyze immediately		
21.	Color	P,G	Cool, 4°C	48 hours		
23–24.	Cyanide, total and amenable to chlorination	P,G	Cool, 4°C, NaOH to pH>12, 0.6g ascorbic acid ⁵	14 days ⁶		
25.	Fluoride	P	None required	28 days		
27.	Hardness	P,G	HNO ₃ to pH<2, H ₂ SO ₄ to pH<2	6 months		
28.	Hydrogen ion (pH)	P,G	None required	Analyze immediately		
31.,43.	Kjeldahl and organicnitrogen	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days		
38.	Nitrate	P,G	Cool, 4°C	48 hours		
39.	Nitrate-nitrite	P,G	Cool, 4°C, H ₂ SO ₄ to pH	28 days		
40.	Nitrite	P,G	Cool, 4°C	48 hours		
41.	Oil and grease	G	Cool, 4°C, HCl or H ₂ SO ₄ to pH<2	28 days		
42.	Organic carbon	G	Cool, 4°C, HCl or H ₂ SO ₄ or H ₃ PO ₄ to pH<2	28 days		
44.	Orthophosphate	P,G	Filter immediately, Cool, 4°C	48 hours		
46.	Oxygen, Dissolved Probe	G Bottle and top	None required	Analyze immediately		
47.	Winkler	G Bottle and top	Fix on site and store in dark	8 hours		
48.	Phenols	G only	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days		
49.	Phosphorus (elemental)	G	Cool, 4°C	48 hours		
50.	Phosphorus, total	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days		
53.	Residue, total	P,G	Cool, 4°C	7 days		
54.	Residue, Filterable	P,G	Cool, 4°C	7 days		
55.	Residue, Nonfilterable (TSS)	P,G	Cool, 4°C	7 days		
56.	Residue, Settleable	P.G	Cool, 4°C	48 hours		
57.	Residue, Volatile	P.G	Cool, 4°C	7 days		
61.	Silica	P, or Quartz	Cool, 4°C	28 days		
64.	Specific conductance	P,G	Cool, 4°C	28 days		
65.	Sulfate	P,G	Cool, 4°C	28 days		
66.	Sulfide	P,G	Cool, 4°C, add zinc acetate plus NaOH to pH >9	7 days		
67.	Sulfite	P,G	None required	Analyze immediately		
68.	Surfactants	P,G	Cool, 4°C	48 hours		
69.	Temperature	P,G	None required	Analyze immediately		
73.	Turbidity	P,G	Cool, 4°C	48 hours		
TABL	E B – Metals ⁷ :					
10.	Boron	P, or Quartz	HNO ₃ to pH<2	6 months		
18.	Chromium VI	P,G	Cool, 4°C	24 hours		
35. & 35m.	Mercury	P,G, or Teflon	HNO ₃ to pH<2	28 days		
71.	Tin	P	HCl or HNO ₃ to pH<2	6 months		

17		Container ¹	echniques, and Holding Times for Wastewater Preservation ^{2,3}	Maximum
Parameter No./name 3, 5–8, 10, 12, 13, Metals:19, 20, 22, 26, 29, (except Cr VI, Sn, Hg, & B)30, 32–34, 36, 37,45, 47, 51, 52, 58–60, 62, 63, 70–72,74, 75.		P,G	HNO ₃ to pH<2	holding time ⁴ 6 months
TABLE	∃ C – Organic Tests ⁸ :		e de la companya della companya della companya de la companya della companya dell	
IA.	Purgeable halocarbons	G, Teflon- lined septum	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵	14 days
IB.	Purgeable aromatics	G, Teflon- lined septum	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵ , HCl to ph<2	14 days
IC.	Acrolein and acrylonitrile	G, Teflon- lined septum	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵ Adjust pH to 4–5 ¹⁰	14 days
II.	Phenois	G, Teflon- lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction; 40 days after extraction
IX.	Benzidines (Benzidine and 3,3-Dichlorobenzidine) ¹¹	G, Teflon- lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days after extraction 13
III.	Phthlate esters ¹¹	G, Teflon- lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
IV.	Nitrosamines ^{11,14}	G, Teflon- lined cap	Cool, 4°C, store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction; 40 days after extraction
V.	PCBs ¹¹	G, Teflon- lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
VI.	Nitroaromatics, cyclic ketones and isophorone ¹¹	G, Teflon- lined cap	Cool, 4° C, store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction; 40 days after extraction
VII.	Polynuclear aromatic hydrocarbons ¹¹	G, Teflon- lined cap	Cool, 4° C, store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction; 40 days after extraction
VIII.	Haloethers ¹¹	G, Teflon- lined cap	Cool, 4° C, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction; 40 days after extraction
IX,	Chlorinated hydrocarbons ¹¹	G, Teflon- lined cap	Cool, 4° C	7 days until extraction; 40 days after extraction
X.	Chorinated Dioxans and Furans	G, Teflon- lined cap	Cool, 4° C, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction; 40 days after extraction

Table F Required Containers, Preservation Techniques, and Holding Times for Wastewater						
Parameter No./name Cont TABLE E Pesticide Tests:		Container ¹ Preservation ^{2,3}	Preservation ^{2,3}	Maximum holding time ⁴		
170.	Pesticides ^{††}		G, Teflon- lined cap	Cool, 4°C, pH 5–9 ¹⁵	7 days until extraction; 40 days after	
1–5.	Alpha, beta, and radium		P,G	HNO ₃ to pH<2	6 months	

¹Polyethylene (P) or Glass (G). For microbiology, plastic sample containers must be made of sterilizable materials (polypropylene or other autoclavable plastic)

²All samples requiring preservation at 4°C must be cooled immediately after collection, and the temperature of the samples shall be documented upon receipt at the laboratory. If the samples are shipped in crushed or cube ice (not "blue ice" packs) and solid ice is still present in the cooler, the lab may simply report the samples as "received on ice". If the ice has melted, the lab must report the either the temperature of the meltwater or of a temperature blank. A temperature blank is defined as an aliquot of deionized water, in an appropriate sample container, which is transported along with the samples. If sampling teams use "blue ice" packs, it is necessary to pre-chill all sample containers to at least 4 degrees celsius with ice or refrigeration prior to shipping. Since shipping simply with "blue ice" packs does not insure that samples are maintained at the appropriate temperatures, the sample collector must submit a temperature blank when using these ice packs for shipping. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting are completed.

³When any sample is to be shipped by common carrier or sent through the United States mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table J, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

⁴Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Virus samples can be stored indefinitely at ~70°C. Samples used for toxicity tests are to be used for test initiation or for renewal of test solutions within 36 hours of collection as grab samples or after removal from composite samplers. For other composite samples, the holding time commences immediately after the samples are removed from the composite sampler. The time the sample spends in the sampler during collection does not count towards the maximum holding time. Samples for biological or chemical analysis may be held for longer periods than specified in this table only if the permittee or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator (s. NR 219.05). Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability.

5Should only be used in the presence of residual chlorine.

⁶Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

⁷Samples should be filtered immediately on-site before adding preservative for dissolved metals.

⁸Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

⁹Samples receiving no pH adjustment must be analyzed within seven days of sampling.

¹⁰The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

¹¹When the extractable analytes of concern fall within a single chemical category, the specified preservation and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidine).

¹²If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 + 0.2 to prevent rearrangement to benzidine.

¹³Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

¹⁴For the analysis of diphenylnitrosamine, add 0.008% Na₂S₂O₃ and adjust pH to 7-10 with NaOH within 24 hours of sampling.

15 The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% Na₂S₂O₃.

NR 219.05 Alternate test procedures. Approvals of alternate test procedures for nationwide use and specific discharges are granted by EPA. The department may approve the use of an alternate test procedure on a case-by-case basis if the crite-

ria for approval of the alternate procedure established in s. NR 149.12 are met. If the department or the EPA approves an alternate test procedure, it shall be considered equivalent to the approved method.

Note: The federal requirements for alternate test procedure approval are given in 40 CFR 136.5.

History: Cr. Register, August, 1976, No. 248, cff. 9–1–76; r. and recr. January, 1978, No. 265, cff. 2–1–78; renum. from NR 219.04 and am. Register, June, 1986, No. 366, cff. 7–1–86; r. and recr. Register, November, 1992, No. 443, cff. 12–1–92; am. Register, February, 1996, No. 482, cff. 3–1–96.

NR 219.06 Laboratory certification or registration.

Bacteriological analyses of groundwater samples, and all radiological analyses shall be performed by the state laboratory of hygiene or at a laboratory certified or approved by the department of health and social services. Other laboratory test results, including effluent toxicity, submitted to the department under a WPDES permit shall be performed by a laboratory certified or registered under ch. NR 149. The following tests are excluded from this requirement:

- (1) Temperature,
- (2) Turbidity,
- (3) Bacteria tests in wastewater effluent and sludges,
- (4) pH,
- (5) Chlorine residual,
- (6) Specific conductance,
- (7) Physical properties of soils and sludges,
- (8) Nutrient tests of soils and sludges,
- (9) Flow measurements.

History: Cr. Register, April, 1986, No. 364, eff. 8-28-86; renum. from NR 219.07 and am. (intro.) Register, November, 1992, No. 443, eff. 7-1-93; am. Register February, 1996, No. 482, eff. 3-1-96.